PARTICLE-SIZE ANALYSIS OF AEOLIAN DUSTS, SOILS AND SEDIMENTS IN VERY SMALL QUANTITIES USING A COULTER MULTISIZER

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ABSTRACT

The Coulter Multisizer has clearly defined strengths and weaknesses as a particle-sizing instrument. It is easier to operate than its Coulter predecessors, though less so than several of its competitors. The Multisizer is best suited to handle very small samples with a narrow particle-size range, such as aeolian dusts and other sediments available only in small quantities. For such samples, Multisizer analysis times are short, resolution is very high (256 size classes) and reproducibility is good. The Multisizer is less well suited to soils and other samples available in large quantities and with a broad particle-size range. For soil particle-size analyses a composite method is proposed involving: Multisizer (2–75 µm), Pipette (<2 µm) and Sieve (>75 µm). © 1997 John Wiley & Sons, Ltd.

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INTRODUCTION

Particle size is a commonly used measure of the physical character of sediments and soils, and a variety of laboratory analysis techniques are available. Significant technological advances have been made in particle sizing and although these have been quickly taken up by manufacturing and mineral processing industries, geomorphologists and other environmental scientists have been slower to exploit this new technology. This paper evaluates the Coulter Multisizer for sizing soils, aeolian dusts and other sediments which are only available in small quantities.

Electrical sensing zone principle of particle sizing

The Multisizer is the latest in a long line of Coulter instruments which have been used in the particle-size analysis of sediments and soils (Walker and Hutka, 1971; McCave and Jarvis, 1973; Walker *et al*., 1974; Shideler, 1976). The Multisizer is based upon the electrical sensing zone (ESZ), or Coulter principle. The number and size of particles is measured by suspending the sample in a conductive liquid and monitoring the electrical current between two electrodes on either side of a small aperture, through which the particles are sucked. As each particle passes through the sensing zone and aperture, it changes the impedance of the current between the two electrodes, producing a pulse with a magnitude proportional to the particle volume. These current pulses are scaled, counted and accumulated in 256 size-related channels from which a particle-size distribution is produced. Miller and Lines (1988) review the ESZ system along with other particle-sizing systems (Figure 1).

ASPECTS OF PARTICLE SIZING BY MULTISIZER

Instrument operation and data output

Ease of operation has not been a strong feature of Coulter instruments in the past and although the Multisizer is a considerable improvement upon earlier Coulter instruments, it still requires more operator experience and

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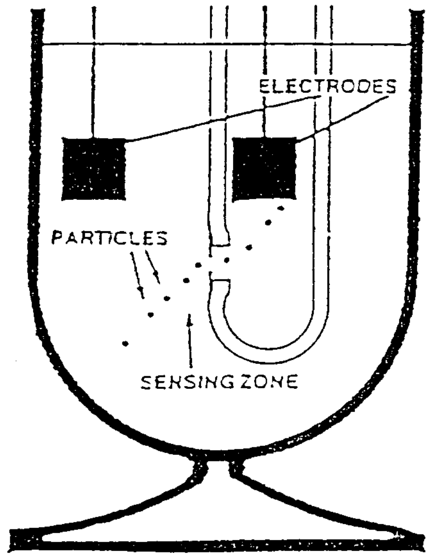


Figure 1. Diagrammatic representation of the electrical sensing zone, or Coulter principle, showing an aperture tube immersed in an electrolyte with particles passing through the aperture (after Coulter Electronics, 1988)

skill than most of its competitors. On the positive side, the Multisizer offers greater potential for operator intervention in analyses, giving the instrument flexibility to handle a broad range of sample types and allowing for inaccuracies to be relatively easily identified and rectified. In terms of data output the Multisizer, like most modern particle-sizing instruments, produces particle-size statistics (mean, median, mode, etc.) but, what is less common, it presents size distributions by volume, number and particle surface area.

Sample pretreatment

As with all particle-size methods, the nature of sample pretreatment has an important control on a particle-size distribution. Analyses performed with and without dispersion, or removal of organic matter, will produce different particle-size distributions. Dust samples collected on glass fibre filter papers require special pretreatment to prevent glass fibres from being sized during an analysis (Kiefert *et al.*, 1992). For soils, dusts and other sediments, dispersion is effected using conventional soil dispersants, such as 10 per cent tri-sodium orthophosphate ($Na_3PO_4.12H_2O$) and 1M sodium hydroxide (NaOH). Undispersed particle-size analyses cannot be performed by Multisizer, as with many other particle-sizing instruments, because the sample must be analysed in a liquid electrolyte. Minimally dispersed analyses can be performed in the electrolyte alone, and provide a useful measure of the size distribution of particles plus stable aggregates. Because of initial dispersion upon wetting, these analyses provide a measure of the aggregates which are stable enough to survive the rigours of sediment transport.

Analysis time

Particle-size analysis of soils and other materials with a broad range of particle sizes can be time-consuming using the Multisizer because the analysis has to be repeated (up to seven times on difficult samples) using different-sized orifice tubes, each covering a portion of the total particle-size distribution. The particle-size distributions from each analysis are then merged using the Coulter AccuComp software. As a general guide, analysis time for a soil sample is comparable with a detailed ($^1/_4\phi$) Pipette–Sieve analysis (McTainsh *et al.*, 1988), and longer than for a Sedigraph. The Multisizer is most time-efficient in analysing samples <100 µm, which is the maximum particle size of most aeolian dusts, particulate air pollutants and other suspended sediments. Such samples can be analysed with two aperture tubes and take <30 min, but this is probably still slower than many laser and light diffraction particle-sizing instruments.

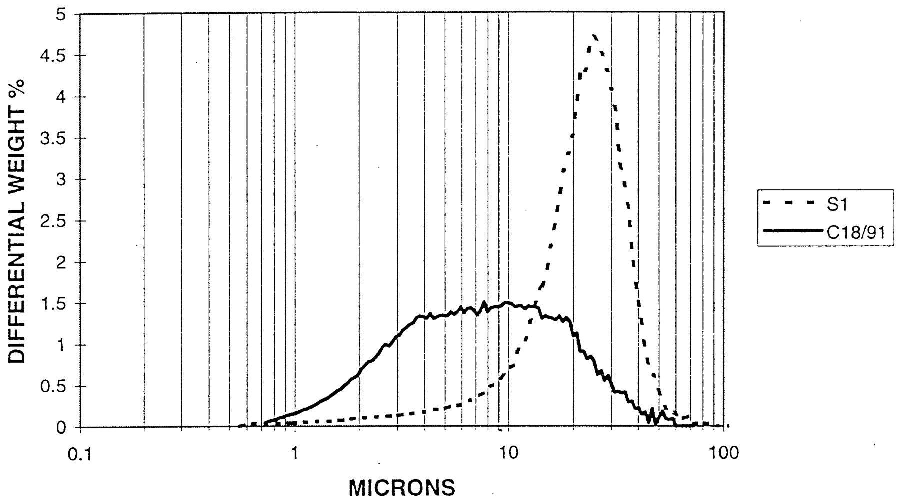


Figure 2. Particle-size distribution (128 size classes) of a suspended dust sampled over 24 days at Charleville, western Queensland (C18/91), and a dust deposit sample collected over 7 h at Sevare, Mali, West Africa (S1)

Sample size

Small sample sizes can pose difficulties for traditional soil particle-size analysis techniques (e.g. Pipette, Hydrometer and Sieve analyses) which require up to 30 g, depending upon the resolution of the analysis. A clear strength of electrical sensing zone instruments is their capacity to analyse samples in very small quantities (Walker *et al.*, 1974; McTainsh and Walker, 1978). This feature has been considerably enhanced in the Multisizer, making it an ideal instrument for studies of wind erosion, aeolian dust transport and dust pollution, in which quantities of trapped sediments are often very low (e.g. <0·001 g) due to naturally occurring low sediment concentrations and low sampling rates of sampling devices, or because only a subsample may be available for particle-size analysis.

The problem of small sample sizes can be reduced in aeolian dust studies by extending sampling periods, but such samples are of less value in dust process studies as, within a long sampling period, wind directions, velocities and other environmental conditions can change, making it difficult to relate sample particle-size characteristics to particular environmental conditions. For example, in Figure 2 sample C18/91 is a suspended dust collected at Charleville, western Queensland, over 24 days, during which time there were two major dust storm events and a prolonged period of background dust. The playkurtic nature of this particle-size distribution reflects the mixing of dusts from several different source areas. Whereas for sample S1 (Figure 2), which is a small dust deposit collected over a period of 7 h by Nickling and Gillies (1991) in Mali, West Africa, the unimodal leptokurtic particle-size distribution can be related to the particular wind speeds, directions and other land conditions at the time of sampling. This deposit was 0·952 g which is too small for some particle-sizing techniques, but was easily analysed with the Multisizer.

Figure 3 shows the particle-size distribution of a very small sample of indoor dust (0·00002 g), the analysis of which would be beyond most particle-sizing instruments. In addition, as the sample was too small to be weighed by a five-decimal-place precision balance, the Multisizer was used to estimate the sample weight. The analysis was performed on the basis of particle counts per millilitre of suspension, to determine the total number of particles and the total volume of particles; then, assuming all particles had a specific density of quartz, the weight was calculated.

Large samples can, however, pose subsampling difficulties for analysis by Multisizer because 0·1 g is an ideal sample size (although this will vary according to particle size), therefore careful subsampling is required to obtain representative subsamples. These subsampling difficulties can be significantly reduced by using a baffled beaker to create a uniform suspension, from which a subsample is taken by pipette. Table I shows the results of replicated particle-size analyses on three baffled beaker subsamples, and although the reproducibility

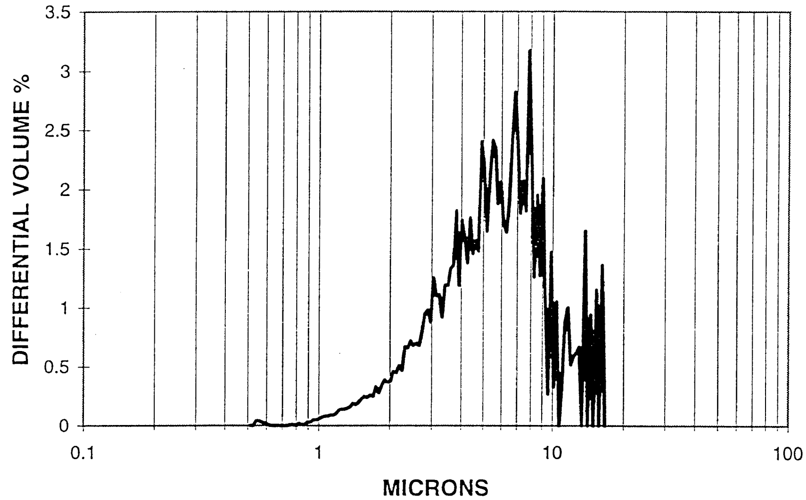


Figure 3. Indoor dust sample collected by low volume air sampler. Total sample weight as measured by counts per millilitre analysis by Multisizer, is 0·00002 g

Table I. Replicated Multisizer analyses (½ϕ intervals) of three subsamples taken from a baffled beaker

Size class: finer than (μm)	Replicated analysis (%) 1	2	3	Mean ± SE
2	11·06	10·83	11·28	11·06±0·13
2·7	15·52	15·16	15·93	15·54±0·22
3·9	21·72	21·09	22·51	21·77±0·41
5·5	28·02	27·24	29·28	28·18±0·59
7·0	32·52	31·83	34·20	32·85±0·70
11·0	41·00	40·58	43·39	41·66±0·88
15·6	46·58	46·34	49·45	47·46±0·99
22·0	53·40	53·61	56·75	54·59±1·08
31·0	60·76	60·75	64·00	61·84±1·08
44·0	72·01	71·00	74·50	72·5±1·04
63·0	89·41	88·75	92·14	90·1±1·04

is very good, there is some loss in precision. The baffled beaker subsampling technique can be scaled to handle samples of <30 g by using larger baffled beakers. Very large dry samples of >30 g can be reduced to a size manageable in a baffled beaker by using a soil-sample splitter.

High-resolution analyses

The Multisizer has few equals for high-resolution particle-size analyses. Particles are sized into 256 size classes (Figure 2 is presented at 128 size classes), whereas some light-scattering instruments (e.g. Coulter LS130) directly size to <100 size classes. In addition, the Multisizer has ‘narrow’ and ‘window’ modes of analysis which allow sizing at 256 size classes over very small size ranges. Figure 4 shows the results of ‘narrow analyses’ of limited size range within the clay fraction (0·45–2 μm) of four soil samples. One sample has a clay mode at 0·78 to 0·8 μm and the other three are finer clays, moding at about 0·58 to 0·62 μm. The three finer distributions are, however, chopped off at 0·5 μm, which is near the limit of the analytical range of the instrument, therefore the actual mode of these clays is probably considerably finer. Although the precise position of the modes in Figure 4 remains in doubt, it is clear that such narrow analyses can discriminate samples and, when combined with clay mineralogical analyses, will help to ‘fingerprint’ soils and sediments to identify sources. High-resolution standard analyses also offer considerable potential for detailed process and provenance interpretations. For example, Figure 5 shows three quite distinct modes at 3, 12 and 40 μm in a

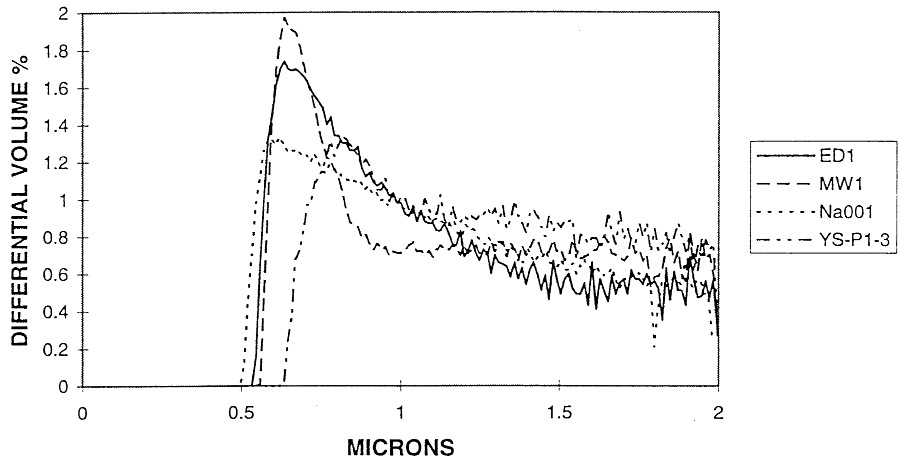


Figure 4. High-resolution particle-size analyses by Multisizer using the 'narrow' mode of analysis on part of the clay fraction (0·45–2μm) of four soil samples

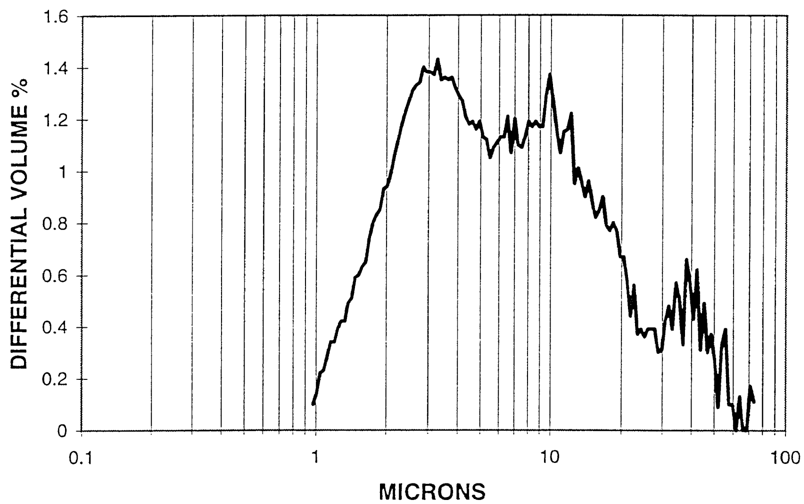


Figure 5. Particle size of a suspended dust (128 size classes) collected in Mali, West Africa, by Nickling and Gillies (1991).

suspended dust sample from Mali. Using the quantum model of Folk (1971), each of these modes represents a log-normally distributed population or quantum of sediments which are from different sources and/or processes. This approach was effective in discriminating dust sources and the role of varying wind conditions in dust deposit formation in Mali (McTainsh *et al.*, 1997).

Analytical range

The Multisizer can measure particles over a large size range (0·45–1200μm), but this involves time-consuming multitube analyses. There are also other practical difficulties with analyses at the coarse end (>150 μm) and the fine end (<2 μm) of the particle-size range.

At the coarse end (>150 μm), the greater mass of the particles makes them more difficult to keep in suspension during an analysis. Use of up to 50 per cent glycerol in a Coulter beaker with baffle, plus a twin-blade stirrer, can enhance suspension, but glycerol is messy to use. Another difficulty arises from low concentrations of coarse particles producing low particle counts, which can be manifest as a jagged tail at the

Table II. Reproducibility of particle-size results from replicated analyses of <2 μm and <20 μm fractions from two soils: Multisizer and Pipette analyses on the same sample

Sample*	%<2 μm Replicates 1	2	3	Mean ± SE	%<22·1 μm Replicates 1	2	3	Mean ± SE
MW1 (MSA)	4·45	4·08	4·25	4·26 ± 0·11	10·17	9·87	10·33	10·12 ± 0·13
YS-P1-3 (MSA)	1·54	1·52	1·56	1·54 ± 0·01	4·68	4·36	4·62	4·55 ± 0·10
MW1 (PA)	14·40	14·49	13·28	14·06 ± 0·38	17·85	18·48	17·41	17·91 ± 0·31

* MSA, Multisizer analysis of sample fraction; PA, Pipette analysis of sample fraction

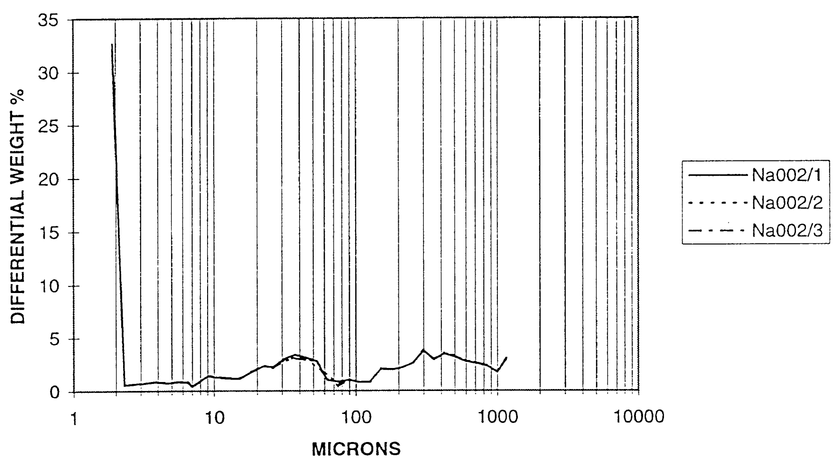


Figure 6. Reproducibility of soil particle-size analyses measured by three replicated analyses using Multisizer (2–75 μm), Pipette (<2 μm) and Sieve (>75 μm)

coarse end of a distribution (Figure 3). Also, the large orifice tubes used for coarse particles consume large quantities of suspension.

While these analytical problems at the coarse end do not preclude Multisizer analyses at an accuracy level at least equal to sieving and at much higher levels of resolution, if sufficient sample is available and high resolution is not required, sieving is a quicker and simpler alternative. A good Multisizer–Sieve method involves wet sieving at 75 μm (near the top end of the 140 μm orifice tube), then dry sieving the >75 μm material using 100 mm diameter Endecott sieves at $^1/_4\phi$ intervals. As both sieving and ESZ sizing techniques are based upon the principle of passing particles through an orifice, there is less of a problem with a 'methodological break' in the analysis than there is with Pipette–Sieve analyses, which combine data from sedimentation and orifice-based sizing techniques in a single particle-size analysis (McTainsh and Duhaylungsod, 1989).

Difficulties also arise with the Multisizer at the fine end of particle-size distributions. Although it provides very detailed data down to 0·45μm (Figure 4), the Multisizer does not measure below 0·45μm, which is one reason why Multisizer measures of clay percentages are often lower than those from sedimentation techniques (Walker and Hutka, 1971) which measure all particles <2 μm. In Table II, the Multisizer measured 4·26 per cent, <2 μm in sample MW1 compared with 14·06 per cent <2 μm by Pipette. This difference is large because sample MW1 has a high proportion of very fine clays (Figure 4) which would not have been measured by the Multisizer. Stein (1985) compared a Sedigraph and a Coulter Counter (a predecessor of the Multisizer) and found that the modal and median particle sizes measured by Sedigraph were lower than that of the Coulter Counter, and this difference increases with decreasing size. Stein suggested that particle shape, specific density and sediment concentration can all affect the measurement of size by sedimentation.

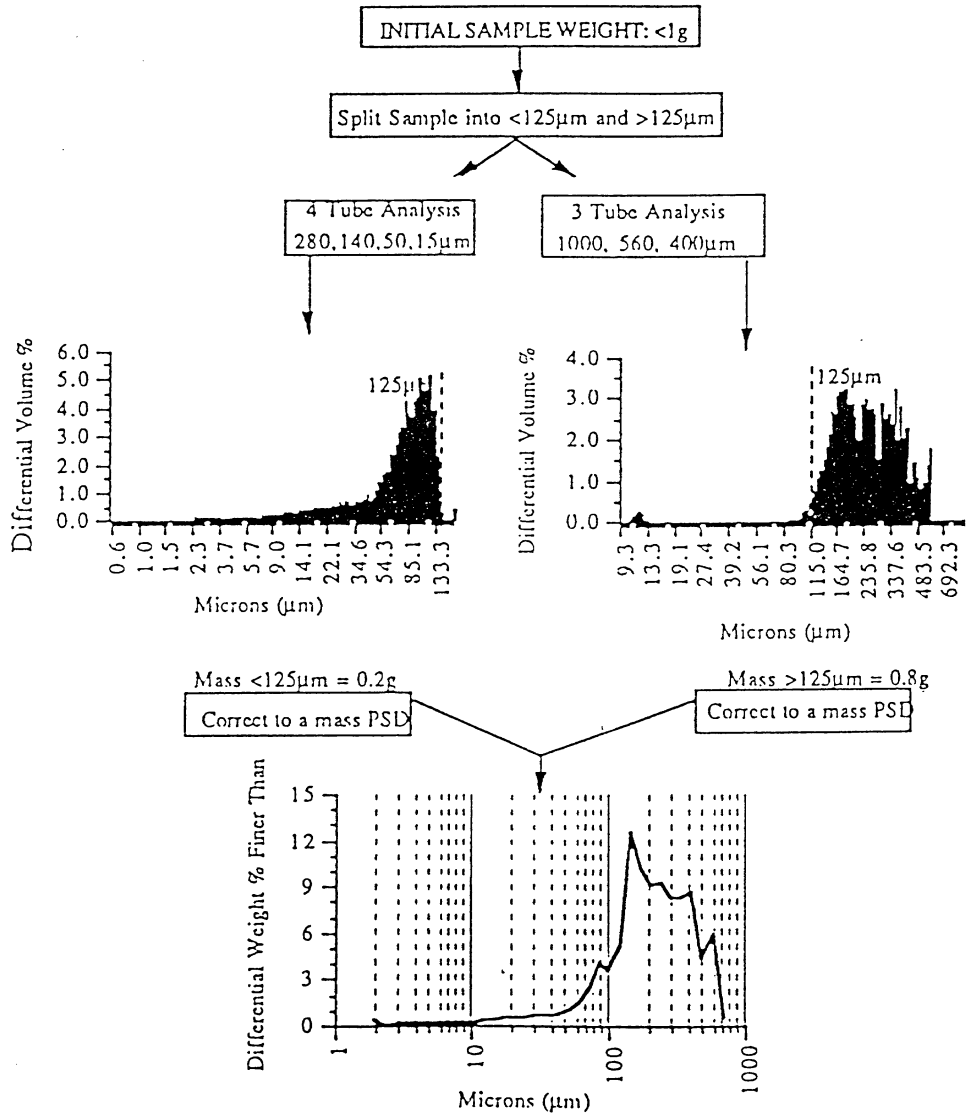
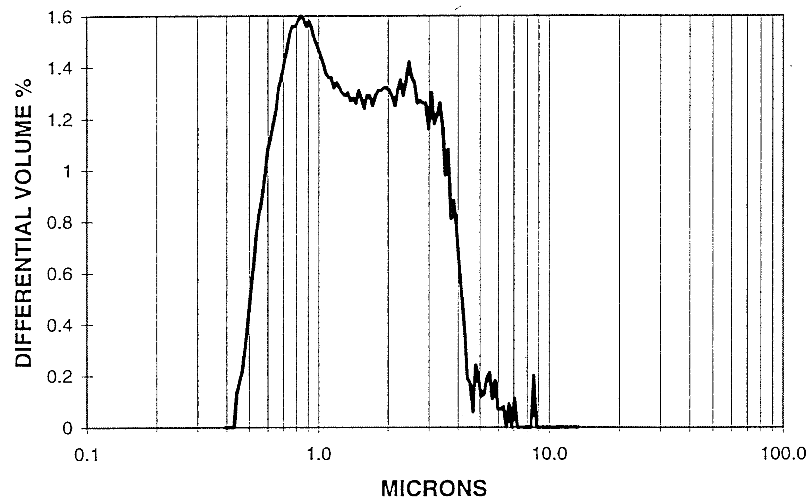


Figure 7. Outline of a method for measuring small samples with a broad particle-size range using a Multisizer

Replicated soil particle-size analyses resulting from Pipette analysis (<2 µm), Multisizer (2 to 75 µm) and Dry Sieve (>75 µm at ¼ϕ intervals) are shown in Figure 6. This method overcomes the problems with the Multisizer at the coarse end (by using sieving) and measures all the material <2 µm (by Pipette), while providing the high resolution of the Multisizer in the silt and fine sand sizes. Analysis times are also faster than for a ½ϕ detailed Pipette/Wet and Dry Sieve analysis technique of McTainsh *et al.* (1988).

Small samples with a broad particle-size range are the most difficult to analyse at high resolution, as there are too few coarse particles to sieve. The Multisizer method summarized in Figure 7 overcomes the practical difficulties at the coarse and fine ends of a distribution by splitting and weighing the sample at 125 µm and analysing the two fractions separately using seven Multisizer tubes in total. Then, using a spreadsheet, the data from two particle-size distributions are combined by recalculating the weight percentages in terms of the total sample weight. This is a time-consuming analysis, but such samples are unable to be analysed by most other particle-sizing techniques.

(a)



(b)

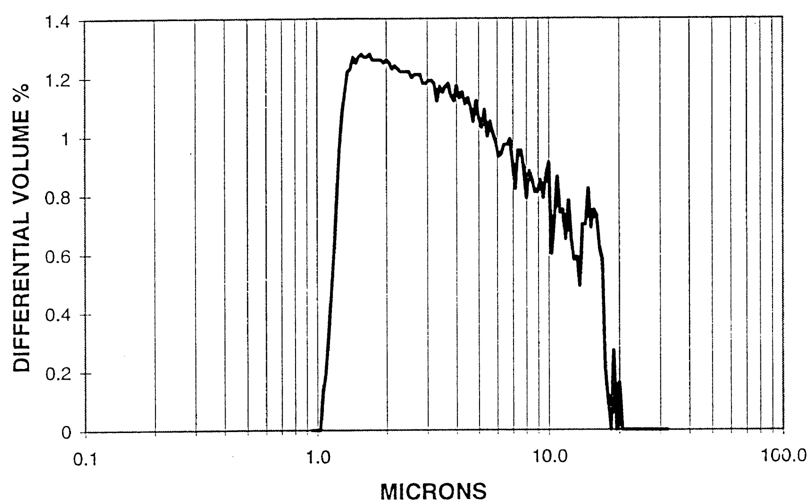


Figure 8. Multisizer analysis of two $^{1}/_{4}\phi$ pipette aliquots: (a) <3·9μm; (b) <15·6μm

Comparison of Pipette and Multisizer

The Multisizer and Pipette methods measure size quite differently – the electrical sensing zone principle versus sedimentation – therefore they would not be expected to produce identical results. Walker and Hutka (1971) compared soil particle-size analyses by Model B Coulter Counter with sedimentation methods, including the standard Pipette method, and concluded that the Coulter Counter compared favourably in terms of efficiency and reproducibility. Given the significant technical advances made since the Model B Coulter Counter, in terms of reproducibility of results, increased resolution and reduced analysis times, we would expect the Multisizer to be much more advanced than Pipette in all respects.

The reproducibility of Multisizer particle-size results is high and is at least comparable with Pipette. Figure 6 shows the minor variation in the appearance of a particle-size distribution resulting from analyses of three replicates by the combined Multisizer–Pipette–Sieve method. Table II shows the reproducibility of three replicated analyses of the clay and silt fractions of two soils, compared with similar analyses by Pipette. Also, Multisizer analyses of pipette aliquots (Figures 8a and 8b) show that the aliquot cutoffs at the coarse ends are reasonably close to where they should be; and, as Figure 9 shows, the particle-size distributions of a soil,

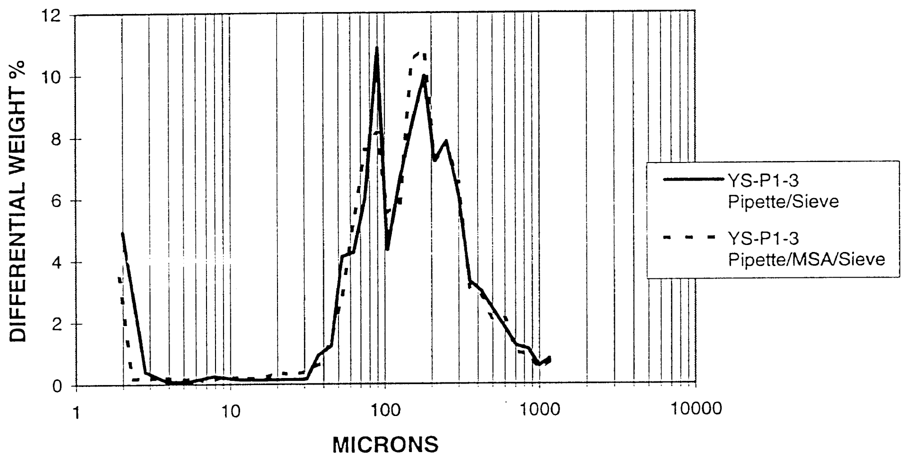


Figure 9. Soil particle-size distributions measured by the Multisizer–Pipette–Sieve method and the detailed Pipette–Sieve method

measured by the detailed Pipette–Sieve method of McTainsh *et al.* (1988) and the Multisizer–Pipette–Sieve method, are similar. The detailed Pipette–Sieve method measures the 2–31 μm fraction by pipette at $^1/_2\phi$ intervals and the >31 μm material is sieved at $^1/_4\phi$ intervals. The pipette data are then expressed as $^1/_4\phi$ equivalents to maintain a constant size class interval throughout the distribution. The results of the Multisizer–Pipette–Sieve method are presented at $^1/_4\phi$ intervals to make them comparable with the detailed Pipette–Sieve analyses, but more detailed levels of data presentation are feasible (e.g. to $^1/_{16}\phi$ size classes or smaller) using equivalent interval values for the sieve fraction.

Although the Pipette method has almost as good reproducibility as the Multisizer, and the particle-size distributions produced by the two methods are similar, only an experienced and skilled Pipette analyst can repeat such results, whereas Multisizer analyses like these can be repeated with little chance of operator error. In addition, most of the errors which arise during a Multisizer analysis are quickly and easily detected before the completion of the analysis and, if necessary, repeat analyses can be quickly performed, whereas errors in Pipette analysis usually do not emerge until after the analysis is completed and the results calculated. There are also differences in the reproducibility of the sieve analyses in Figures 6 and 9. This difference may be because 10 cm diameter sieves were used for the analysis in Figure 6, whereas 20 cm diameter sieves were used in Figure 9. It is likely that sample contamination and/or loss would have been greater with the larger diameter sieves.

Putting aside the inconsistencies in the dry sieve analyses, the Multisizer–Pipette–Sieve method is faster, capable of much higher resolution, and has better reproducibility than the detailed Pipette–Sieve method. However, for routine batch analyses at low resolution (e.g. percentage sand, silt and clay), Pipette analyses would be much faster.

SUMMARY OF CONCLUSIONS

1. The Multisizer is easier to operate than its Coulter predecessors, though less so than several of its competitors, with the exception of detailed Pipette–Sieve analyses which require considerable experience and skill. Data output includes particle-size statistics plus volume, number and surface-area distributions.
2. For samples with a narrow particle-size range, Multisizer analysis times are short and comparable with other instruments, but for soils the Multisizer is still relatively slow.
3. The Multisizer is well suited to handle very small samples, which makes it very suitable for analysing aeolian dusts and similar sediments. Analyses of large samples are made difficult by the need to representatively split the sample down to about 0·1 g for analysis.
4. The Multisizer sizes at 256 size class intervals, which is a much higher resolution than most other particle-sizing instruments. Very detailed analyses can be performed using the ‘narrow’ and ‘window’ modes of

analysis, which allow 256 size class analyses over a small particle-size range. For small samples with a broad particle-size range, the Multisizer has few competitors.

5. The reproducibility of Multisizer results is high.
6. The analytical range of the Multisizer is greater than that of other Coulter instruments, but analyses >150 μm can be difficult and time-consuming. The lower limit of analytical range is 0·45μm and, unlike sedimentation techniques, no measurements finer than this can be made. Clay percentages measured by Multisizer are consistently lower than by the Pipette method.
7. For soil particle-size analyses a composite method is proposed involving Multisizer (2–75 μm), Pipette (>2 μm) and Dry Sieve (>75 μm). When compared with the detailed Pipette–Sieve method for soil analyses, the Multisizer is faster, has higher resolution and higher reproducibility.

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